

Los Alamos National Laboratory  
Environmental Restoration Program  
Standard Operating Procedure

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Sample Containers and Preservation

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## **Sample Containers and Preservation**

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## **Sample Containers and Preservation**

### **1.0 PURPOSE**

This procedure describes specific requirements for sample containers, preservation techniques and holding times as specified by field regulations and guidance documents.

### **2.0 SCOPE**

#### **2.1 Applicability**

This procedure is applicable to all Environmental Restoration (ER) activities involving the collection and preservation of samples that will be shipped to the Los Alamos National Laboratory Sample Coordination Facility (SCF) for subsequent chemical or physical testing.

#### **2.2 Training**

The Field Team Leader (FTL) is responsible for the implementation of this procedure and the FTL and field team members must document that they have read and understand this procedure and the other procedures in Section 1.0, General Instructions.

### **3.0 DEFINITIONS/ACRONYMS**

- A. ASTM: American Society for Testing and Materials
- B. CLP: Contract Laboratory Program
- C. DOT: U.S. Department of Transportation
- D. EPA: U.S. Environmental Protection Agency
- E. Holding time: The elapsed time between sample collection and initiation of laboratory analysis. The holding time is from date of sampling to date of analysis, not the date the designated laboratory receives the sample.
- F. IATA: International Air Transport Association
- G. RCRA: Resource Conservation and Recovery Act
- H. RFI: RCRA Facilities Investigation
- I. SCF: Sample Coordination Facility
- J. SW-846: EPA approved test methods for Solid Waste identified in EPA-SW-846.

- K. TCLP: Toxicity Characteristic Leaching Procedure (Method 1311), which is a codified (40CFR Parts 261, 264, 265, 268, 271, and 302) procedure.

#### **4.0 BACKGROUND AND/OR CAUTIONS**

The use of specific types of sample container and preservation techniques is mandatory for hazardous site investigations because the integrity of any sample is diminished over time. Physical factors (light, pressure, temperature, etc.), chemical factors (changes in pH, volatilization, etc.), and biological factors may alter the original quality of the sample. Because the various target parameters are uniquely altered at varying rates, distinct sample containers, preservation techniques, and holding times have been established to maintain sample integrity for a reasonable and acceptable period of time.

The volume of sample collected should be sufficient to perform all the required analyses, plus an additional amount to provide for any quality control needs, split samples, or repeat examinations. The volumes, preservatives, and holding times listed in Attachments A through F are the EPA requirements. Since the SCF operated by EM-9 will be either performing the analyses or making arrangements for the analyses, sampling schedules and sample needs must be coordinated with the SCF prior to sampling.

All proposed ER Program sampling plans are reviewed at the SCF for adherence to sampling protocols mandated by all applicable EPA regulations and analytical methods. The Sampling and Analysis Plan (SAP) will address the proper analytical protocol. SW-846 provides test procedures and guidance which are recommended for use in conducting the evaluations and measurements needed to comply with the RCRA (Public Law 94-850). These methods are approved by EPA to satisfy the data requirements of 40 CFR 120-270. The topics of concern include the sampling schedule, proper sample sizes and containers, correct preservation techniques, chain-of-custody requirements, and transportation of samples to the SCF. Appendix O of the Los Alamos National Laboratory Installation Work Plan details the duties of the SCF.

#### **5.0 EQUIPMENT**

Equipment needed to implement this procedure is listed on the Equipment and Supplies Check List for Sample Containers and Preservation (Attachment G).

#### **6.0 PROCEDURE**

- A. Contact the SCF for guidance and assistance in obtaining the proper sample containers and preservatives.
- B. Refer to an Operable Unit (OU)-specific SAP for site-specific procedures for sample containers and preservation. In addition, follow the protocols established in EPA's SW-846, Test Methods for Evaluation of Solid Waste.

- C. Record all pertinent comments and any deviations on the Daily Activity Log per LANL-ER-SOP-01.04.
- D. Contact the SCF for guidance and acquisition of the proper sample containers.
  - 1. Identify the containers required for analysis by matrix as shown in Attachments A through F (for example, amber glass, narrow-mouth bottles for polychlorinated biphenyl [PCB] analysis of water samples).
    - a. Based on information in the SAP, choose a sample container that is nonreactive with the sample and the particular analytical parameter to be tested.
    - b. Use glass or fluorocarbon resin containers with samples analyzed for organic compounds to prevent the introduction of extraneous organic compounds, such as those that might be leached from plastic containers.
    - c. The rigid plastic screw caps for the bottles must be Teflon lined to prevent contamination of the sample.
  - 2. Calculate and order the number of each type of container required by including duplicates and blanks with the number of investigative samples specified in the OU-specific SAP.
  - 3. Acquire a sufficient number of containers to ship the proper sample volume. For example, Department of Transportation (DOT) and International Air Transport Authority (IATA) regulations limit the size of a sample container to 16 oz if the contents may include hazardous materials. In this case, two 500-ml or four 250-ml containers would be required to ship a 1-liter fluid sample.
  - 4. Adhere to DOT regulations for on-site transfer of samples to the SCF over public-access roads. Refer to SOP-01.03, Handling, Packaging, and Shipping of Samples, for additional information.

## **6.1 Containing Procedures**

- A. If soil, sludge or wastes are submitted to the laboratory in containers other than those in which they were collected, the container should be glass, wide mouth bottles with a Teflon cap liner. For specific methods, see Attachment D.
- B. If soil samples are submitted to the laboratory in the sample equipment in which they were collected, the container must have the end covered with Teflon or aluminum foil, have a plastic cap placed over the end, and have the cap taped in place. This procedure reduces the chance for loss of moisture or volatile organic compounds (VOCs).

- C. The type and size of container used for water samples and aqueous waste samples varies based on the type of analysis to be performed. Refer to Attachments A and B for specific information on water sample containers and Attachment C for information on aqueous waste sample containers.
- D. Sample containers received from SCF will be precleaned to EPA specifications before they are sent to the site. A certificate of cleanliness will be retained by the SCF, with a copy provided with the containers.

## **6.2 Sample Preservation**

- A. Regardless of the sample type or container, unless site-specific guidelines state otherwise, samples collected for chemical analysis should be preserved by placing them in an insulated container (cooler) and maintained on ice (ice in bags or chemical "blue" ice) at 4° Centigrade (C).
- B. Avoid freezing the sample by wrapping it in bubble pack to isolate from the "blue" ice.
- C. Containers for water samples and aqueous waste may arrive at the OU site from the SCF with the proper type and amount of preservatives in them. If so, do not prerinse or overfill.
- D. If on-site preservation of aqueous samples is necessary, the proper reagents should be provided to field team members in an easily usable form that can be added at the time of sampling. Reagents that are designated as hazardous by the DOT Hazardous Materials Table, Title 49 Code of Federal Regulations (CFR) 172.101, are shipped pursuant to the appropriate DOT regulations.
- E. If using an acid or base preservative, check the pH adjustment with pH paper.
- F. Preservation required for the specific analyses requested for aqueous samples may be determined by using Attachments A through C, or by consulting the referenced documents.
- G. Solid sediment and solid waste for chemical analysis will always be preserved on ice to 4°C. See Attachments D through F.
- H. A sufficient amount of ice should be brought to the field to ensure proper sample cooling immediately following collection. A temperature of 4°C must be maintained until the samples arrive at the SCF.
- I. The temperature shall be checked and recorded at the laboratory to determine if preservation has been accomplished.

### **6.3 Holding Time**

- A. Analyze samples as soon as possible after collection.
- B. Some parameters are required to be analyzed in the field (refer to Attachment A).
- C. Allowable holding times are listed and are the maximum times that samples are considered valid, based on available guidance (see Attachments A through F).
- D. The OU-specific maximum holding times will be listed in the RFI Workplan.
- E. Holding times for SW-846 methods start when the sample is collected. Both the sampler and the laboratory use this time/date.
- F. Holding times for the Contract Laboratory Program (CLP) methods for the samples is the day of collection. The laboratory date is date received at the designated laboratory. Delayed shipping will result in missed holding time for the data. Transfer the samples to the SCF as soon as possible.

### **7.0 REFERENCES**

The following procedures are directly associated with this procedure and should be reviewed before field operations commence:

LANL-ER-SOPs in Section 1.0, General Instructions  
LANL-ER-SOP-02.07, General Equipment Decontamination  
LANL-ER-SOP-06.03, Sampling for Volatile Organics

Title 40 CFR Part 261

Title 49 CFR Part 172.101

EPA (U.S. Environmental Protection Agency), 1982. "Handbook for Sampling and Sample Preservation of Water and Wastewater." Report EPA-600/4-82-029. Washington, D.C.

EPA (U.S. Environmental Protection Agency), 1983. "Methods for Chemical Analysis of Water and Wastes." Report EPA-600/4-79-020. Washington, D.C.

EPA (U.S. Environmental Protection Agency), 1983. "Manual of Groundwater Quality Sampling Procedures." Report EPA/600/2-81-160. Washington, D.C.

EPA (U.S. Environmental Protection Agency), 1986. "Test Methods for Evaluating Solid Waste." Report EPA-SW-846. Washington, D.C.

EPA (U.S. Environmental Protection Agency), 1985. "Practical Guide for Groundwater Sampling." Report EPA/600/2-85/104. U.S. Government Printing Office, Washington, D.C.

EPA (U.S. Environmental Protection Agency), 1986. "RCRA Groundwater Monitoring Technical Enforcement Guidance Document." Document OSWER-9950.1. U.S. Government Printing Office, Washington, D.C.

EPA (U.S. Environmental Protection Agency) Region IV, 1991. "Environmental Compliance Branch Standard Operating Procedures and Quality Assurance Manual," Appendix A, Environmental Services Division, Athens, Georgia

Korte, Nic, and Peter Kearl, 1985. "Procedures for the Collection and Preservation of Groundwater and Surface Water Samples and for the Installation of Monitoring Wells: Second Edition." U.S. Department of Energy Report GJ/TMC-08 Technical Measurements Center, Grand Junction Project Office, Grand Junction, Colorado

Williams, M.C., 1990. Handbook for Sample Collection, Preservation, Instrumental Techniques. Los Alamos National Laboratory Report LA-11738-M, Los Alamos, New Mexico

## **8.0 RECORDS**

- A. Completed Chain-of-Custody/Request for Analysis Form
- B. Completed Daily Activity Log

## **9.0 ATTACHMENTS**

- A. Recommendation for Sampling and Preservation of Water Samples According to Measurement (Low- to Medium-Contaminant Concentration Samples)
- B. Sampling and Preservation Procedures for RCRA Groundwater Detection Monitoring
- C. Containers for Aqueous Waste Samples
- D. Preservation Plan for Soil/Sediment Samples
- E. Sample Containers for Waste
- F. Preservation Plan for Soil/Sediment Waste Samples
- G. Equipment and Supplies Check List for Sample Containers and Preservation



**RECOMMENDATION FOR SAMPLING AND PRESERVATION OF  
WATER SAMPLES ACCORDING TO MEASUREMENT<sup>(a)</sup>  
(LOW TO MEDIUM CONTAMINANT CONCENTRATION SAMPLES)**

Measurement	Volume Requirement millimeter (ml)	Container <sup>b,c</sup>	Preservative <sup>d,e</sup>	Holding Time <sup>f,g</sup>
<u>Physical Properties</u>				
Color	50	P,G	Ice <sup>h</sup>	48 hrs
Specific Conductance	100	P,G	Ice	24 hrs
Hardness	100	P,G	HNO <sub>3</sub> to pH<2	6 mos
Odor	200	G only	Ice	24 hrs
pH	25	P,G	N/A	Determine on site
Residue				
Filterable	100	P,G	Ice	7 days
Nonfilterable	100	P,G	Ice	7 days
Total	100	P,G	Ice	7 days
Volatile	100	P,G	Ice	7 days
Settleable Matter	1000	P,G	Ice	48 hrs
Temperature	1000	P,G	N/A	Determine on site
Turbidity	100	P,G	Ice	48 hrs

Metals (except Mercury and Chromium<sup>+6</sup>)

Dissolved	200	P,G	Filter on site HNO <sub>3</sub> to pH<2	6 mos 6 mos <sup>i</sup>
Suspended	200	P,G	Filter on site	6 mos
Total	100	P,G	HNO <sub>3</sub> to pH<2	6 mos
<u>Chromium<sup>+6</sup></u>	200	P,G	Ice	24 hrs

Mercury

Dissolved	100	P,G	Filter HNO <sub>3</sub> to pH<2	28 days
Total	100	P,G	HNO <sub>3</sub> to pH<2	28 days

**RECOMMENDATION FOR SAMPLING AND PRESERVATION OF  
WATER SAMPLES ACCORDING TO MEASUREMENT<sup>(a)</sup>  
(LOW TO MEDIUM CONTAMINANT CONCENTRATION SAMPLES)  
(Continued)**

Measurement	Volume Requirement (ml)	Container <sup>b,c</sup>	Preservative <sup>d,e</sup>	Holding Time <sup>f,g</sup>
<u>Inorganics, Nonmetallics</u>				
Acidity	100	P,G	Ice	14 days
Alkalinity	100	P,G	Ice	14 days
Bromide	100	P,G	None Required	28 days
Chloride	100	P,G	None Required	28 days
Chlorine	200	P,G	N/A	Determine on site
Cyanides	500	P,G	Cool 4°C Ice NaOH to pH>12 0.6 g ascorbic acid	14 days
Fluoride	300	P,G	Ice	28 days
Iodide	100	P,G	Ice	24 hrs
Nitrogen				
Ammonia	400	P,G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Nitrogen, Total Kjeldahl	500	P,G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Nitrate Plus Nitrite	100	P,G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Nitrate <sup>h</sup>	100	P,G	Ice	48 hrs
Nitrite	50	P,G	Ice	48 hrs
<u>Dissolved Oxygen</u>				
Probe	300	G bottle and top	N/A	Determine on site
Winkler	300	G bottle and top and store in dark	Fix on site	8 hrs
<u>Phosphorus</u>				
Orthophosphate, Dissolved	50	P,G	Filter on site, Ice	48 hrs
Hydrolyzable	50	P,G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days

**RECOMMENDATION FOR SAMPLING AND PRESERVATION OF  
WATER SAMPLES ACCORDING TO MEASUREMENT<sup>(a)</sup>  
(LOW TO MEDIUM CONTAMINANT CONCENTRATION SAMPLES)  
(Continued)**

Measurement	Volume Requirement (ml)	Container <sup>b,c</sup>	Preservative <sup>d,e</sup>	Holding Time <sup>f,g</sup>
Total, Dissolved	50	P,G	Filter on site, Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	24 hrs
Silica	50	P only	Ice	28 days
Sulfate	50	P,G	Ice	28 days
Sulfide	50	P,G	Ice 2 ml zinc acetate(2N) NaOH to pH >9	24 hrs
Sulfite	100	P,G	N/A	Determine on site
<u>Organics</u>				
BOD	1,000	P,G	Ice	48 hrs
COD	50	P,G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Oil and Grease	1,000	P,G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Organic Carbon	25	P,G	Ice H <sub>2</sub> SO <sub>4</sub> or HCl to pH<2	28 days
Phenolics	500	G only	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Cyanides	1,000	P,G	Ice 40% NaOH to pH>12 0.6 g ascorbic acid	14 days
Coliform, Fecal, and Total	250	P,G	Ice Sterile	6 hrs
Oil and Grease	1,000	G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Organic Carbon	25	P,G	Ice H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Phenols	1,000	G, Teflon™-lined cap	Ice	7 days until extraction; 40 days after extraction
Total Organic Halogen and Purgeable Aromatics	40	G, vial with Teflon™-lined septum	Ice 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>j</sup>	14 days

**RECOMMENDATION FOR SAMPLING AND PRESERVATION OF  
WATER SAMPLES ACCORDING TO MEASUREMENT<sup>(a)</sup>  
(LOW TO MEDIUM CONTAMINANT CONCENTRATION SAMPLES)  
(Continued)**

Measurement	Volume Requirement (ml)	Container <sup>b,c</sup>	Preservative <sup>d,e</sup>	Holding Time <sup>f,g</sup>
Purgeable Aromatics	40	G, vial with Teflon <sup>TM</sup> -lined septum	Ice 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>k</sup>	14 days
Acrolein and Acrylonitrile	1,000	G, Teflon <sup>TM</sup> -lined septum	Ice 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>k</sup>	14 days
Phenols	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice	7 days until extraction; 40 days after extraction
Benzidines	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>k</sup>	7 days until extraction; 40 days after extraction
Phthalate Esters	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>k</sup>	7 days until extraction; 40 days after extraction
Nitrosamines	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice store in dark 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>k</sup>	7 days until extraction; 40 days after extraction
Nitroaromatics and Isophorone	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice	7 days until extraction; 40 days after extraction
Haloethers	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice	7 days until extraction; 40 days after extraction
Chlorinated Hydrocarbons	1,000	G, Teflon <sup>TM</sup> -lined cap	Ice	7 days until extraction; 40 days after extraction

**RECOMMENDATION FOR SAMPLING AND PRESERVATION OF  
WATER SAMPLES ACCORDING TO MEASUREMENT<sup>(a)</sup>  
(LOW TO MEDIUM CONTAMINANT CONCENTRATION SAMPLES)  
(Continued)**

Measurement	Volume Requirement (ml)	Container <sup>b,c</sup>	Preservative <sup>d,e</sup>	Holding Time <sup>f,g</sup>
Tetrachlorodibenzo-p-dioxin	1,000	G, Teflon™-lined cap	Ice	7 days until extraction; 40 days after extraction
<u>Pesticides and PCBs</u>				
Pesticides and PCBs	1,000	G, Teflon™-lined cap	Ice	7 days until extraction; 40 days after extraction
<u>Radiological Tests</u>				
Alpha, Beta and Radium	1,000	P,G	HNO <sub>3</sub> to pH<2	No limit
Tritium	25	G	None	No limit
Isotopic Uranium	500	P	HNO <sub>3</sub> to pH<2	No limit
<sup>90</sup> Sr	1,000	P	HNO <sub>3</sub> to pH<2	No limit
<sup>238</sup> Pu	500	P,G	HNO <sub>3</sub> to pH<2	No limit
<sup>239</sup> Pu	500	P,G	HNO <sub>3</sub> to pH<2	No limit
<sup>137</sup> Cs	500	P,G	HNO <sub>3</sub> to pH<2	No limit
<sup>241</sup> Am	500	P,G	HNO <sub>3</sub> to pH<2	No limit

<sup>a</sup>More specific instructions for preservation and sampling are found with each applicable SAP. A general discussion about sampling water and industrial wastewater is found in EPA Region IV (1991); EPA (1986); and EPA (1985).

<sup>b</sup>Container Types:

P = Plastic (polyethylene)

G = Glass

T = Fluorocarbon resins (PTFE, Teflon™, FEP or PFA)

PP = Polypropylene

<sup>c</sup>Shipping containers (cooling chest with ice or ice pack) should be certified as to the 4°C temperature at the time of sample placement into these containers. Preservation of samples requires that the temperature of collected samples be cooled to 4°C upon collection of sample and during shipment. Field personnel will check the temperature in the container at the time of shipping and ice the samples to maintain a cool temperature during shipment. Maximum-minimum thermometers can be placed into the shipping chest to record temperature history. Chain-of-custody forms will include the temperature in the container at the time of shipment and at delivery to the laboratory in addition to in-transit (maximum) temperature, if available.

<sup>d</sup>Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, samples may be preserved by cooling to 4°C until compositing and splitting are completed.

<sup>e</sup>When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the DOT Materials Regulations (49 CFR Part 172). The SCF is responsible for ensuring compliance. For preservation requirements, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following material: Hydrochloric acid (HCl) in water solutions at concentrations of 0.22% by weight or less, with pH less than 2.0 but greater than or equal to 1.0.

<sup>f</sup>Samples should be analyzed as soon as possible after collection. The times listed are the maximum periods that samples may be held and still be considered valid. Samples may be held for longer periods only if the permittee, or analytical laboratory, has data on file to show that the specific types of samples under study are stable for a longer time and has received a variance from the EPA Regional Administrator.

<sup>g</sup>Maximum holding time is 24 hours when sulfide is present. Optional testing of all samples with lead acetate paper before the pH adjustment will determine if sulfide is present. If sulfide is present, it can be removed by adding of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and the pH is adjusted to 12 with NaOH.

<sup>h</sup>Shipping containers (cooling chest with ice or ice pack) should be verified as to the 4°C temperature at the time of sample placement into these containers. Preservation of samples requires that the temperature of collected samples be cooled to 4°C upon collection of sample and during shipment. Field personnel will check the temperature in the container at the time of shipping and ice the samples to maintain a cool temperature during shipment. Chain-of-custody forms will include the temperature in the container at the time of shipment and at delivery to Laboratory.

<sup>i</sup>Samples should be filtered on site immediately, before adding preservative for dissolved metals.

<sup>j</sup>For samples from nonchlorinated drinking water supplies, concentrated  $\text{H}_2\text{SO}_4$  should be added to lower sample pH to less than 2. The sample should be analyzed within 14 days.

<sup>k</sup>Should only be used in the presence of residual chlorine.

<sup>l</sup>Use ml volume requirements for water and gram requirements for media other than liquids.

<sup>m</sup>No preservation for media other than water.

## SAMPLING AND PRESERVATION PROCEDURES FOR RCRA GROUNDWATER DETECTION MONITORING<sup>a</sup>

Parameter	Minimum Volume Required for Analysis (ml)	Recommended Container <sup>b,c</sup>	Preservative <sup>d,e</sup>	Maximum Holding Time <sup>f,g</sup>
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### Indicators of Groundwater Contamination<sup>h</sup>

pH	25	T,P,G	Field determined	None
Specific Conductance	100	T,P,G	Field determined	None
Total Organic Carbon	4 x 15	G, amber, T-lined cap <sup>f</sup>	Ice	28 days HCl to pH<2
Total Organic Halogen	4 x 15	G, amber, T-lined septa or caps	Ice, add 1.1M sodium sulfite	7 days

### Groundwater Quality Characteristics

Chloride	50	T, P, G	Ice	28 days
Iron	200	T, P	Field acidified to pH <2 with HNO <sub>3</sub>	6 months
Manganese				
Sodium				
Phenols	500	G	Ice/H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days
Sulfate	50	T, P, G	Ice	28 days

### Drinking Water Characteristics

Arsenic	500	T, P	<u>Total Metals</u> Field acidified to pH <2 with HNO <sub>3</sub>	6 months
Barium				
Cadmium				
Chromium	500			6 months
 <u>Dissolved Metals</u>				
Lead		T,P		
Mercury	500	T,P	Field filtration	6 months
Selenium		T,P		
Silver		Dark Bottle	Acidify to pH <2 with HNO <sub>3</sub>	



**SAMPLING AND PRESERVATION PROCEDURES FOR RCRA GROUNDWATER  
DETECTION MONITORING<sup>a</sup> (Continued)**

Parameter	Minimum Volume Required for Analysis (ml, unless noted otherwise)	Recommended Container <sup>b,c</sup>	Preservative <sup>d,e</sup>	Maximum Holding Time <sup>f,g</sup>
Fluoride	300	T, P	Ice	28 days
Nitrate/Nitrite <sup>i</sup>	1,000	T, P, G	Ice/H <sub>2</sub> SO <sub>4</sub> to pH <2	14 days
Endrin	2,000	T, G	Ice	7 days
Lindane				
Methoxychlor				
Toxaphene				
2,4 Dichlorophenol				
2,4,5 Trichlorophenol				
Silvex				
Radium	1 gallon	P, G	Field acidified to	6 months
Gross Alpha		pH <2 with HNO <sub>3</sub>		
Gross Beta				
Coliform bacteria	200	PP, G (sterilized)	Ice	6 hours

Other Groundwater Characteristics of Interest<sup>h</sup>

Cyanide	250	P, G pH >12. 0.6 g ascorbic acid <sup>j</sup>	Ice, NaOH to	14 days
Oil and Grease	1,000	G only pH <2	Ice H <sub>2</sub> SO <sub>4</sub> to	28 days
Semivolatile, Nonvolatile Organics	60	T, G	Ice	14 days
Volatiles	60	G, T-lined	Ice	14 days

<sup>a</sup>References:

EPA (U.S. Environmental Protection Agency) 1986. "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods," Report EPA-SW-846 (3rd edition). Washington, D.C.

EPA (U.S. Environmental Protection Agency) 1983. "Methods for Chemical Analysis of Water and Wastes," Report EPA-600/4-79-020. Washington, D.C.

"Standard Methods for the Examination of Water and Wastewater," 16th edition (1985).

**<sup>b</sup>Container Types:**

P = Plastic (polyethylene)

G = Glass

T = Fluorocarbon resins (PTFE, Teflon™, FEP or PFA)

PP = Polypropylene

**<sup>c</sup>Shipping containers (cooling chest with ice or ice pack) should be certified as to the 4°C temperature at the time of sample placement into these containers. Preservation of samples requires that the temperature of collected samples be cooled to 4°C upon collection of sample and during shipment. Field personnel will check the temperature in the container at the time of shipping and ice the samples to maintain a cool temperature during shipment. Maximum-minimum thermometers can be placed into the shipping chest to record temperature history. Chain-of-custody forms will include the temperature in the container at the time of shipment and at delivery to the laboratory in addition to in-transit (maximum) temperature, if available.**

**<sup>d</sup>Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, samples may be preserved by cooling to 4°C until compositing and splitting are completed.**

**<sup>e</sup>When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the DOT Materials Regulations (49 CFR Part 172). The SCF is responsible for ensuring compliance. For preservation requirements, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following material: Hydrochloric acid (HCl) in water solutions at concentrations of 0.22% by weight or less, with pH less than 2.0 but greater than or equal to 1.0.**

**<sup>f</sup>Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before the pH adjustment to determine if sulfide is present. If sulfide is present, it can be removed by adding of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then the pH is adjusted to 12 with NaOH.**

**<sup>g</sup>Based on the requirements for detection monitoring (40 CFR 265.93), the owner/operator must collect a sufficient volume of ground water to allow for the analysis of four separate replicates.**

**<sup>h</sup>Do not allow any headspace in the container.**

**<sup>i</sup>For samples from nonchlorinated drinking water supplies, concentrated H<sub>2</sub>SO<sub>4</sub> should be added to lower sample pH to less than 2. The sample should be analyzed within 14 days.**

**<sup>j</sup>Use ascorbic acid only in the presence of oxidizing agents.**

### CONTAINERS FOR AQUEOUS WASTE SAMPLES<sup>a</sup>

Analyte	Sample Container	Sample Volume	Preservative <sup>b,c</sup>	Holding Time(days)
Hazardous Substance List (HSL) Volatile	G vials (2)	40 ml	Ice	14
HSL Base/Neutral/Acid <sup>d</sup>	Amber G	1 l	Ice	7/40 <sup>e</sup>
HSL Pesticide/PCB	Amber G	1 l	Ice	7/40 <sup>e</sup>
HSL Inorganic <sup>f</sup>	P,G	1 l	pH<2,w/HNO <sub>3</sub>	180
Non-HSL Metals <sup>g</sup>	P,G	1 l	pH<2,w/HNO <sub>3</sub>	180
Cyanide	P,G	0.5 l	pH>11,w/NaOH	14
pH <sup>h</sup>	P,G	N/A	None	N/A
Specific Conductivity <sup>h</sup>	P,G	N/A	None	N/A
Temperature <sup>h</sup>	P,G	N/A	None	N/A
Dissolved Oxygen <sup>h</sup>	G	N/A	None	N/A
Total Dissolved Solids (TDS)	P,G	0.1 l	Ice	7
Total Suspended Solids (TSS)	P,G	0.1 l	Ice	7
Total Phosphate	P,G	1 l	Ice, pH<2, w/H <sub>2</sub> SO <sub>4</sub>	28
Chloride, Sulfate	P,G	1 l	Ice	28
Carbonate/Bicarbonate <sup>i</sup>	P,G	1 l	Ice	14
Nitrate	P,G	1 l	Ice	2
Hexavalent Chromium	P,G	1 l	Ice	24

<sup>a</sup>Reference:

1. EPA (U.S. Environmental Protection Agency) 1986. "Test Methods for Evaluating Solid Waste," Report EPA SW-846 (3rd edition). Washington, D.C.

<sup>b</sup>All samples, with the exception of those for total metals, will be filtered within four hours of sample collection and preservatives will be added to the filtrate as specified.

<sup>c</sup>All samples will be kept at 4°C after arrival at the Laboratory.

<sup>d</sup>The HSL base/neutral/acid fractions analytical parameters are the HSL semivolatiles.

<sup>e</sup>Extract within 7 days; analysis within 40 days of extraction.

<sup>f</sup>Includes cesium, molybdenum, and strontium, which are non-HSL metals, but are analyzed using the same methods.

<sup>g</sup>Includes Target Analyte List (TAL) Metals

<sup>h</sup>Field measurements.

<sup>i</sup>These are reported as carbonate and biocarbonate alkalinity.

## PRESERVATION PLAN FOR SOIL/SEDIMENT SAMPLES

Analyte	Sample Volume (mL)	Sample Container <sup>f</sup>	Method	Preservative	Holding Time (days)
Volatile Organics	5	40-ml vial (2) with Teflon <sup>TM</sup> -lined silicon rubber septum	Ref. 2	Ice	14
Base/Neutral/Acid Extractable Organics	10-30	Amber G, 1 l	Ref. 3	Ice	7/40 <sup>a</sup>
Organochlorine Pesticide/PCB	10-30	Amber G, 1 l	Ref. 4	Ice	7/40 <sup>a</sup>
HSL Inorganic <sup>b</sup>	200	P, G, 1 l	Ref. 5	Ice	180
Non-HSL Metals <sup>c</sup>	200	P, G, 1 l	SW-846	Ice	180
Reactivity	---	Amber G	Ref. 6	Ice	N/A
Chloride	20	G, 1 l	EPA 300.0 <sup>d</sup>	Ice	N/A
Sulfate	20	G, 1 l	EPA 300.0 <sup>d</sup>	Ice	N/A
Nitrate	20	G, 1 l	EPA 300.0 <sup>d</sup>	Ice	N/A
Cyanide	200	G, 1 l	Ref. 1	Ice	14
Hexavalent Chromium	100	G, 1 l	S.M. 312b <sup>e</sup>	Ice	1

<sup>a</sup>Extract within 7 days; analysis within 40 days of extraction.

<sup>b</sup>Includes cesium, molybdenum, and strontium, which are non-HSL metals, but are analyzed using the same methods.

<sup>c</sup>Includes Target Analyte List (TAL) metals

<sup>d</sup>Soil/sediments will be leached with laboratory reagent water (20 grams (g) soil to 50 ml water) and water extract will be analyzed using the procedure in "Methods for Chemical Analysis of Water and Wastes," 1983; EPA 600/4-79-020.

<sup>e</sup>Soil/sediment will be leached with laboratory reagent water (5 g soil and 100 ml of water) by shaking for 2 hours, and the water extract will be filtered and subsequently analyzed. This is in accordance with method 312B in Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985.

<sup>f</sup>Container types:

P = Plastic (polyethylene)

G = Glass

### **Method References**

Ref. 1. Method 9010 - "Test Methods for Evaluating Solid Wastes," EPA report EPA-SW-846, Office of Solid Waste and Emergency Response, Washington, D.C. 20460, Revised September 1986.

Ref. 2. Method 8240 - "Test Methods for Evaluating Solid Wastes," EPA report EPA-SW-846, Office of Solid Waste and Emergency Response, Washington, D.C. 20460, Revised September 1986.

Ref. 3. Method 8270 - "Test Methods for Evaluating Solid Wastes," EPA report EPA-SW-846, Office of Solid Waste and Emergency Response, Washington, D.C. 20460, Revised September 1986.

Ref. 4. Method 8080 - "Test Methods for Evaluating Solid Wastes," EPA report EPA-SW-846, Office of Solid Waste and Emergency Response, Washington, D.C. 20460, Revised September 1986.

Ref. 5. Method 6010 or 7000 Series Methods - "Test Methods for Evaluating Solid Wastes," EPA report EPA-SW-846, Office of Solid Waste and Emergency Response, Washington, D.C. 20460, Revised September 1986.

Ref. 6. Method 9010 or 9030 Series Methods - "Test Methods for Evaluating Solid Wastes," EPA report EPA-SW-846, Office of Solid Waste and Emergency Response, Washington, D.C. 20460, Revised September 1986.

### SAMPLE CONTAINERS FOR WASTE<sup>a</sup>

Waste Type	Recommended Container	Closure	Analysis
Photosensitive wastes: 1,000 or 2,000 ml	Amber HDPE <sup>b</sup> or amber glass caps with Teflon <sup>TM</sup> liners for glass bottles	Teflon <sup>TM</sup> caps for HDPE bottles; Bakelite	Waste characterization per 40 CFR Part 261
Pesticides: hydrocarbon chlorinated; hydrocarbons; petroleum distillates	Wide-mouth, borosilicate glass bottles 1,000 or 2,000 ml	Bakelite caps with Teflon <sup>TM</sup> liner	Waste characterization per 40 CFR Part 261
Oil wastes	HDPE bottles, wide mouth, 1,000 or 2,000 ml	Teflon <sup>TM</sup> caps	Waste characterization per 40 CFR Part 261
Strong alkali or hydrofluoric acid	HDPE bottles, 1,000 ml	Teflon <sup>TM</sup> caps, wide mouth	Waste characterization per 40 CFR Part 261
Aqueous waste--characterization of organics	Borosilicate glass bottles 1,000 or 2,000 ml	Caps with Teflon <sup>TM</sup> liner	Waste characterization per 40 CFR Part 261
Solids (sludge, soils, and granular)	8-oz wide-mouth glass bottle	Bakelite caps with Teflon <sup>TM</sup> liners	Waste characterization per 40 CFR Part 261

#### <sup>a</sup>References

1. Title 40 CFR Part 261.
2. EPA (U.S Environmental Protection Agency) 1986. "Test Methods for Evaluating Solid Waste," Report EPA SW-846 (3rd edition). Washington, D.C.

<sup>b</sup> High Density Polyethylene (HDPE).

## PRESERVATION PLAN FOR SOIL/SEDIMENT WASTE SAMPLES

Analyte	Sample Container	Sample Volume	Preservation	Holding Time (days)
HSL Volatile	40 ml vials (2)	5 g	Ice	14
TCLP Volatile	40 ml vials (2)	5 g	Ice	14
HSL Base/Neutral/Acid <sup>a</sup>	Amber G	10-30 g	Ice	7/40 <sup>b</sup>
TCLP Semivolatiles	Amber G	10-30g	Ice	7/40 <sup>b</sup>
HSL Pesticide/PCB	Amber G	10-30 g	Ice	7/40 <sup>b</sup>
TCLP Pesticides/Herbicides	Amber G	10-30g	Ice	7/40 <sup>b</sup>
HSL Inorganic <sup>c</sup>	P,G	200 g	Ice	180
Non-HSL Metals <sup>d</sup>	P,G	200 g	Ice	180
TCLP Metals	P,G	200g	Ice	180
Reactivity	Amber G	100 g	Ice	N/A
EP Toxicity	Amber G	100 g	Ice	N/A
Chloride	G <sup>e</sup>	20 g	Ice	N/A
Sulfate	G <sup>e</sup>	20 g	Ice	N/A
Nitrate	G <sup>e</sup>	20 g	Ice	N/A
Cyanide	G	200 g	Ice	14
Hexavalent Chromium	P,G <sup>f</sup>	1,000 g	Ice	24

<sup>a</sup>The HSL base/neutral/acid fractions analytical parameters are the HSL semivolatiles.

<sup>b</sup>Extract within 7 days; analysis within 40 days of extraction.

<sup>c</sup>Includes cesium, molybdenum, and strontium, which are non-HSL metals, but are analyzed using the same methods.

<sup>d</sup>Includes Target Analyte List (TAL) Metals

<sup>e</sup>Soil/sediments will be leached with laboratory reagent water (20 g soil to 50 ml water) and water extract will be analyzed using referenced procedure. Procedure references: "Methods for Chemical Analysis of Water and Wastes," 1983; EPA 600/4-79-020.



<sup>f</sup>Soil/sediment will be leached with laboratory reagent water (5 g soil and 100 ml of water) by shaking for 2 hours. The water extract is filtered and subsequently analyzed. This is in accordance with method 312B in "Standard Methods for Examination of Water and Wastewater," 15th Edition, 1985

## **EQUIPMENT AND SUPPLIES CHECK LIST FOR SAMPLE CONTAINERS AND PRESERVATION**

### Forms

- \_\_\_\_\_ Sample Collection Logs
- \_\_\_\_\_ Daily Activity Log
- \_\_\_\_\_ Chain-of-Custody/Request for Analysis Form

### Sample Containers, as appropriate

- \_\_\_\_\_ Narrow-mouth amber glass bottles with Teflon™-lined caps (0.5, 1, and 2 liters)
- \_\_\_\_\_ Amber glass vials with Teflon™ septa (40 ml)
- \_\_\_\_\_ 250-ml sterile bottle
- \_\_\_\_\_ Wide-mouth polyethylene bottles (0.5, 1, and 2 liters)
- \_\_\_\_\_ New or cleaned polyethylene narrow-mouth bottles (1 l, 10 l, 500 ml, 125 ml, and 60 ml)

### Sampling Materials

- \_\_\_\_\_ Ballpoint pen (indelible dark ink)
- \_\_\_\_\_ Felt-tip marker pen (indelible dark ink)
- \_\_\_\_\_ 1-14 pH indicator paper
- \_\_\_\_\_ Ascorbic acid crystals
- \_\_\_\_\_ Disposable surgical gloves (latex, PVC, other suitable plastic, or rubber)
- \_\_\_\_\_ NaOH pellets
- \_\_\_\_\_ Disposable wipes
- \_\_\_\_\_ Crystalline  $\text{Na}_2\text{S}_2\text{O}_3$
- \_\_\_\_\_ Methanol and deionized water in Teflon™ wash bottles

**EQUIPMENT AND SUPPLIES CHECKLIST  
FOR SAMPLE CONTAINERS AND PRESERVATION (Continued)**

- \_\_\_\_\_ Concentrated HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, and HCl
- \_\_\_\_\_ Temperature probe
- \_\_\_\_\_ Clipboards
- \_\_\_\_\_ Deionized water
- \_\_\_\_\_ Duct tape
- \_\_\_\_\_ Wooden tongue depressors
- \_\_\_\_\_ Aluminum foil
- \_\_\_\_\_ Teflon™ tape
- \_\_\_\_\_ Paper towels

**Shipping Materials (acquire from Sample Coordinator Facility)**

- \_\_\_\_\_ Cardboard boxes
- \_\_\_\_\_ Ice
- \_\_\_\_\_ Blue Ice or equivalent
- \_\_\_\_\_ Insulated coolers
- \_\_\_\_\_ Heavy-duty poly bags and ties
- \_\_\_\_\_ Strapping tape
- \_\_\_\_\_ Plastic trashcan liners
- \_\_\_\_\_ Canvas bags
- \_\_\_\_\_ Parafilm
- \_\_\_\_\_ Padding for packaging of samples
- \_\_\_\_\_ Ziplock® bags
- \_\_\_\_\_ Bubble pack
- \_\_\_\_\_ Unique sample sticker sheets
- \_\_\_\_\_ Chain-of-Custody/Request for Analysis forms

**EQUIPMENT AND SUPPLIES CHECKLIST  
FOR SAMPLE CONTAINERS AND PRESERVATION (Continued)**

- \_\_\_\_\_ Sample labels
- \_\_\_\_\_ Custody seals or custody tape
- \_\_\_\_\_ Other equipment specified in EPA Methods, as needed